

# PATENT ABSTRACTS OF JAPAN

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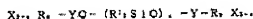
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## (54) AQUEOUS SILICONE EMULSION WHICH FORMS SILICONE ELASTOMER HAVING IMPROVED ADHESION TO SUBSTRATE

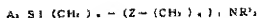
(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an aqueous silicone emulsion having improved storage stability and adhesion by mixing water with a surfactant, a crosslinking agent, a tin condensation catalyst, a specified diorganosiloxane polymer, a specified amino-functional siloxane and an acid.

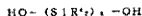
SOLUTION: A diorganosiloxane polymer represented by formula I [wherein (n) is 0-3; (z) is 200-15; X is hydroxyl or a hydrolyzable group; R is a 1-5C (un)substituted monovalent hydrocarbon group; R1s are each X or R, provided that at least 90% of them are Rs; Y is Si, -Si-(CH2)mSiR12-group, -Si-(CH2)m-SiR12-SiR12-(CH2)mSiR12-group and (m) is a positive integer] is mixed with water, a surfactant, a crosslinking agent, a tin condensation catalyst, an amino-functional siloxane prepared by reacting an amino-functional silane represented by formula II [wherein A is a hydrolyzable group; Z is O or NR2; R2 is H or a 1-15C (un)substituted monovalent hydrocarbon group; (p) and (q) are each 2-10; and (r) is 0-3] with a hydroxyl-terminated organosiloxane represented by formula III (wherein R4 is R; and (b) is 4-80) and an acid.



I



II



III

**\* NOTICES \***

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1. This document has been translated by computer. So the translation may not reflect the original

precisely.

2 \*\*\* shows the word which can not be translated.

3. In the drawings, any words are not translated.

## CM ATMS

**[Claim(s)]**

[Claim 1] JIOTRUGANO siloxane polymer (I) which is a drainage system silicone emulsion which forms a silicone elastomer which has an improved adhesive property to a substrate when water is removed, and is shown with a silicone emulsion (A) following formula containing the following

$X_{n-1}R_n-VO-(R^1SiO)_z-VX_{n-1}$  — here,  $n$  is 0, 1, 2, or 3, and  $z$  is an integer of 200–10,000.  $X$  is a hydroxylated SHIKUR group or a certain hydrolytic basis, and  $R^1$  individually, 1–15 carbon atoms were released, or it is an unsaturated univalent hydrocarbon group.  $R^1$  is individually chosen from  $X$  basis

and R group, however at least 90% of  $R^1$  is R group, Y And an Si atom and  $-Si(CH_2)_m-SiR^1_2$ -basis, Or (B) water whose m it is a  $-Si(CH_2)_m-SiR^1-O-SiR^1_2-SiR^1_2$ -basis, and it is as having

defined  $R^1$  here above, and is a positive integer;

(C) A surface-active agent;

(D) Arbitrary cross linking agents;

(E) A tin condensation catalyst

(F) Amino functionality Silang (II) shown with a following formula

A Si(CH<sub>3</sub>)<sub>2</sub>-Z-(CH<sub>2</sub>)<sub>n</sub>-NR<sup>3</sup>. (here) A is a hydrolytic basis and Z is an oxygen atom or NR<sup>2</sup>

[illegible]

[[Translation done]]









[0071] Since the performance characteristics of a silicone elastomer formed from a silicone emulsion or of a certain kind is affected, additional arbitrary ingredients, for example, a filler, and other ingredients may be added with other ingredients according to a wish. As an example of a reinforcing filler and a filler for increase in quantity, there are calcium carbonate, a titanium dioxide, a zinc oxide, iron oxide, and kaolin clay. As an example of a filler which can be used in order to give fire retardancy or electric arc resistance, there are aluminum  $\beta$  hydrate, a fumed titanium, and way acid zinc. Other arbitrary ingredients which include paints, stabilizer, or reinforcement nature resin in the spot can be added to this invention silicone emulsion.

[0072] An ingredient of an addition of all these should be examined in order that they may secure not having bad influence on the adhesive property of a silicone elastomer and storage life which are said formed when a silicone emulsion or this emulsion of this invention is dried. The character of the said drying system silicone emulsion and said silicone elastomer can give the characteristics of a wish by making it change and changing arbitrary ingredients of these particles of a particle size, a particle size of a silicone improved adhesion to a substrate and this resin. A particle size seems however, to exist in the range of 100-5000 nm diameter of silicone emulsion. This time is usually several months but/or several seasons of length when particle size of this emulsion is formed by various methods. For example, this silicone emulsion of this invention is formed by various methods. For example, this silicone emulsion can be prepared by a method of an emulsion polymerization currently taught to US-A

2891102, 3294725, 3355403, 3380491, and 3897469.

[0073] About an emulsion polymerization, an emulsion of linear siloxane oligomer is underwater distributed with a surface-active agent, and a reserve mixture is formed. Although an amphiphilic surface active agent, an anionic surfactant, or a cationic surfactant is generally used, a mixture of an amphiphilic surface active agent, an anionic surfactant or a cationic surfactant, and a nonionic surface active agent will also function. Subsequently, this reserve mixture is mixed by high shearing until an emulsion containing a disperse phase containing a glob of a with aqueous phase and a particle size of 100-5000 nm silicone oligomer is formed. This mixture may take place also in what kind of type of commercial mixing device, and said mixed device is well-known at a person skilled in the art. In order to adjust pH, acid or a base may be added to said emulsion, or it may be added to a reserve mixture. Instead, said surface-active agent may be converted into a form of the acid or a base using an ionic exchange process as taught by US-A 3697469. Although a polymerization catalyst is used, a saturation at the room temperature, it may carry out at a high temperature and a high pressure, and requirements are 25 °C - 80 °C. Probably, generally the polymerization is 1 to 24 hours depending on temperature and a molecular weight of a with a polymer. After, JORUGANO siloxane polymer reads a molecular weight of a with a polymerization is suspended by neutralizing this emulsion.

[0074] A cross linking agent for [being required] — or — if wished) and a tin condensation catalyst [0075] can be added before emulsification or after a polymerization. However, a cross linking agent and a tin condensation catalyst will often be added to an emulsion, after a polymerization finishes. This cross linking agent shifts into a disperse phase from aqueous phase in this case, and, in addition, must maintain that reactivity.

[0076] After said amino functionality siloxane is added at any [ between emulsification polymerizations ] time, for example, a reserve mixture as some reserve mixtures before emulsification is emulsified, a front stirrup of a polymerization is added behind. As for it, when being added before said amino functionality siloxane's emulsifying, adding within 8 hours is preferred.

[0077] In a comparatively low-concentration polymer solid content is desired, water of an addition amount may be added in which stage of combination. A general polymer solid content is 20 to 75%. A desirable polymer solid content is 40 to 75%.

[0078] This acid may be independently added as a part of other ingredients, after a polymerization is completed. As a part of other ingredients, DDDTA will form acetic acid, for example, when it adds to water.

[0080] Arbitrary ones of other ingredients, for example, a filler, paints, stabilizer, reinforcement nature resin in the spot, etc. can be added to any time, after a polymerization is completed.

[0081] A desirable method of preparing a drainage of a silicone emulsion is directly based on an emulsification method, and this is well-known and is taught to a person skilled in the art at US-A

http://www4.ipdl.jpdl.jp/ep-bin/ran\_web.cgi?atw=HtUp3A3Z5F2Wf4IodL. 2010/04/30

4171717 or EP-A 0739947, 0739922, and 0739923.

[0082] A mixture of JORUGANO siloxane polymer by which performing was carried out, a surface-active agent, and water is emulsified sufficient period and by mixing by heating, JORUGANO siloxane polymer is characterized as what has the viscosity 5000 - 50000 mPa·s at 25 °C. However, supposing viscosity is prepared using a solvent, a polymer blend, etc., polymer of a comparatively high molecular weight can be used. A surface active agent, an anionic surfactant, a cationic surfactant, or a nonionic surfactant is used as independent or a mixture. This mixing takes place in a commercial mixing device of one of types. The mixed device is well-known at a person skilled in the art.

[0084] A cross linking agent (when required, or when it is wished) and the tin condensation catalyst can add a front stirrup of emulsification after emulsification. However, said cross linking agent and a tin condensation catalyst will often be added before emulsification. If it adds after emulsification, this cross linking agent must shift into a disperse phase from water, and must still maintain that reactivity.

[0085] Said amino functionality siloxane can be added even in front of emulsification or in the back at any time. When being added after this amino functionality siloxane's emulsifying, it is preferred to add within 8 hours after bridge construction of an emulsion.

[0086] When a low polymer solid content is desired, water of an addition amount can also be added in one stage of the combination. A general polymer solid content is 10 to 95%. A desirable polymer solid content is 20 to 85%, and is much more desirable, 1 to 40 to 85% of it. [0087] Acid can also be added at any time as a part of other ingredients (namely, DDDTA will form acetic acid at the time of addition of water). In a desirable method, carboxylic acid and an amino functionality siloxane are added before bridge construction. If this wishes, it will make it possible to reduce quantity of a cross linking agent and a tin condensation catalyst.

[0088] Any arbitrary agent can be added to an emulsion, after a polymerization is completed. It will make it possible to add a filler, paints, stabilizer, resin for reinforcement in the spot, etc.

[0089] In a more desirable method, it is JORUGANO siloxane polymer (here) of 100 weight sections.

X is hydroxy, n is 2 and Y is Si, R and R' is a methyl group, respectively — an amino functionality siloxane, hydroxy siloxane, and a catalyst 1 weight section of polyoxyethylene alkyl ether surface-active agent 0.053 weight section [ water:2 copy ] (this) Amino functionality Silane  $(\text{O}(\text{CH}_2)_2)_2\text{Si}-\text{CH}_2-\text{NH}(\text{CH}_2)_2\text{NH}_2$ , hydroxy and ORGANO siloxane  $(\text{H}(\text{HO})-\text{Si}(\text{CH}_3)_2)_2\text{O}$  (here) it is formed by making Silane (IV) methyl trimethoxysilane react, and b is 1-3. And an amino functional group of 0.005 weight sections exists, and a mole ratio of ingredient (II) and ingredient (IV) opposite ingredient (III) is at least 1. Again, a mole ratio of ingredient (IV) pair ingredient (II) is two or less, and acetic acid of 0.1 copy under high shearing, and form a high solid concentration emulsion, and it dilutes so that this high solid concentration emulsion may be dispersed in water. Subsequently by constructing a bridge in this emulsion, a drainage of 0.9 weight sections.

[0090] After removing water, a siloxane emulsion is formed from this invention silicone emulsion has an improved adhesive property to a substrate. A silicone emulsion of this invention has long storage life with the conventional emulsion.

[0091] [Example] The following examples are shown in order to explain the constituent of this invention further. In the following examples, unless it refused in particular, one day after making an emulsion, the cast of the humid emulsion was carried out to the film, and it examined, after drying these films for seven days.

[0092] The result of the durometer was obtained by the method indicated to ASTM C961 "Indentation Hardness of Elastomer-Type Sealant Means of a Durometer". The result of C961 "Indentation Hardness of Elastomer-Type Sealant Means of a Durometer" which is 121 mm, it obtained by the tension and elongation. The size of L, the dumbbell specimen which is 121 mm, it obtained by the method indicated to "Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers-Tension" which used and was indicated to ASTM D412. The Shore A hardness value was obtained by the method indicated to ASTM C961 "Indentation Hardness of Elastomer Type Sealant by Means

http://www4.ipdl.jpdl.jp/ep-bin/ran\_web.cgi?atw=HtUp3A3Z5F2Wf4IodL. 2010/04/30

of a Diuron<sup>®</sup> (0.04837 M, and 50% of relative humidity, <sup>a</sup> "Et" means ethyl, and "laboratory conditions" is temperature  $\pm 3 \pm 3^\circ\text{C}$ ), and used the adhesive valuation method sample was formed on various substrates to the bead (0.04941 M) in length, and 18 mm in width. The adhesive property was evaluated by putting eluting (based) 50 mm in length, and pulling this adhesive, and pulling this bead by hand at the end using a laser blade by sealant / substrate interface, and a bead caused cohesive failure, the into 45 degrees to a level base material surface. When a bead caused cohesive failure, the adhesive property was graded "easy". The bead caused adhesive failure, and when power equivalent to the time of removing a bead from a base material surface was required, the adhesive property was graded with "good". When adhesive failure was caused by the corollation power in which a bead is comparatively low, it graded with "++".

(0.0951) Example 1) Hydroxy and polydimethyl siloxane (PDMS) polymer of 5000 copies (viscosity is 50 Pa.s at 25  $^\circ\text{C}$ ). The 10:90, Turullo, mixer was loaded with the Tergitol(trademark) TMAN-dissolution (0.001) water of 6 (s is ethylation time) trimethyl nonanol with a nonionic surface active agent), and 100 copies of 100 copies.

[0060] The mixer was stirred under the vacuum for 5 minutes using the distributed brad revolving at 1200 rpm. After mixing when it observed, the emulsion was not homogeneous and the gel (CHMSO TO rope) of non fluidity, and formation of the c/w emulsion of high solid concentration was shown. The distributed brad was rotated at 600 rpm, stirring was begun, and DI water of 250 copies was added. The distributed brad applying a vacuum and rotating the distributed brad at 1200 rpm — an emulsion — further — it stirred for 2 minutes. When observed, the white emulsion looked like milk this time. The distributed brad was rotated at 600 rpm, stirring was begun again, and DI water of 1000 copies was divided into two equal portions, and was added to the emulsion. This time, the mixture was stirred during [2 minute] and this had the same color. To the emulsion mixed that it is white and like milk was depressed, and this had neither a lump nor gel. To the emulsion, in the hydroxy and PDMS fluid (the forms of the polymerization) — a weight of 25 or 0.4 P-4 or 74.6 copies, the N-2'-aminoethyl- $\gamma$ -aminopropyl trimethoxysilane (AEATPS) of 152 copies, and the methyl TORMETOSIL vinyl (MTM) of 10.2 copies, the distributed micro functionally siloxane (AF2) of 50 copies was added by carrying out cold blending. The distributed mixer was rotated at 600 rpm, stirring of this mixture was started, the blending. The applied, and this mixture was stirred for 2 minutes, subsequently — adding BTMS (butyltrimethoxysilane) of 40 copies — the bottom of a vacuum — a mixture — further — it (bottom 4 min) was stirred of 40 copies. The distributed mixer was added and this mixture was stirred for 2 minutes, using the distributed brad rotated at 600 rpm under a vacuum. The Sameco (trade mark) cartridge was filled up with this silicone emulsion that constructed the bridge, and all the air, which centrifuged this and was caught was removed.

[illegible]

[0000] —  
the same conclusion. I also I agree on separate issues.

**Table 1**  
Example of sealant adhesiveness substrate (Silicone PlusGE II, ceramic tile A, \*\* A DETURANA Duranor) \*\*, Good Right concrete A Good Right Korean Corian), \*\* Good An A paint spreading pine (aluminum paint), A An A paint spreading pine (flax patch) A A United States Japan cedar A A A alkyd aluminum A A A polvinyl chloride (unplasticized) A Good A glass A Good A brass A A \*\*\*\* A A polycarbonate \*\* Good \*\* stainless steel A Right mortar A Good Right PMMA (acrylic methacrylate) \*\*, Good \*\* #10093 (Example 2)

polymer of 50 Ppm, and one copy of amino functionality siloxane (AF72) prepared in Example 1, and stirred it for 30 seconds under the vacuum. Subsequently, DETDA of 0.06 g was added and the mixture was stirred for 30 more seconds under the vacuum. Glacial acetic acid of 0.1 g was added and the mixture was stirred for 30 more seconds under the vacuum. Next, Tetraol (trademark) TMH-6 (the notation is omitted) of 0.1 g, the ethylenediamine poly (nonanol) and DI water of three copies (6 the notation is omitted) were added, and this mixture was stirred for 30 more seconds under the vacuum, and the mixture of high solid content of a little transparent non fluidity was obtained. Subsequently, a sample of 100 g of four copies each, adding BTMS of 0.5 g — this mixture — further — for 30 seconds. The Sarnex (trademark) cartridge was filled up with this silicone emulsion, and all the air which centrifuged this was removed. After aging at a room temperature for 18 hours, the sample was formed on the 2.54 mm (100 mils) thick film, and it was made to harden under the laboratory condition for 40 days. The tensile strength of this hardened translucent silicone latex elastomer was 0.25N/da (40 psi), 1570% of the maximum elongation, and the Shore A durometer 6. On the glass as a substrate, concrete, the pine that carried out the painting color, and a U.S. Japan cedar, other samples of the silicone emulsion aged for 18 hours were formed to a bead 90 mm in length, and 18 mm in width, and will be stiffened under laboratory conditions for 14 days. As the adhesive property of said silicone latex was indicated above (A), it was evaluated; said latex was excellent in all the substrates — i.e. +++++++(ad) (cohesive failure). After carrying out storage aging of said damp silicone emulsion for four weeks under a laboratory condition, as the sample of other silicone emulsions was shown above (A), it was examined about the adhesive property. Said latex showed the adhesive property excellent in all the substrates (cohesive failure).

[0100] (b) Amino functionality siloxane was not added at all, and also the same procedure as [0100] (a) was followed. The tensile strength of this hardened translucent latex elastomer was 0.26N/da (38 psi), 1005% of the maximum elongation, and the Shore A durometer 11. This elastomer showed the +\*\*\* adhesive property to glass, and showed the good storage aging property to concrete, the paint spreading pine, and the U.S. Japan cedar. After aging the storage aging of said damp silicone emulsion for four weeks under a laboratory condition, as the sample of further others of the above-mentioned emulsion was indicated above (A), it was examined about the adhesive property. This elastant showed the +\*\*\* adhesive property to glass, and showed the good adhesive property to paint spreading pine, and the U.S. Japan cedar.

(a) The tensile strength of this hardened translucent silicone elastomer was 0.32MPa (46 psi), 1280% of the maximum elongation, and the Shore A durometer 6. This elastomer showed the adhesive property outstanding to glass, concrete, the paint spreading pin, urethane, etc. After carrying out storage aging of said damp silicone emulsion for four weeks under laboratory condition, it still maintained its original adhesive properties without change in color, appearance, or about the sample of further others of said damp silicone emulsion was indicated as shown in Figure 1(A). It also showed good adhesion to plastic, metal, paper, and other materials, and especially to glass, and showed the good adhesiveness properties.

(b) The second adhesive preparation was made by adding 0.0152 copy of the APS to the APS solution, and stirred for 10 seconds in the Hauschild™ D102 mixer at 95 rpm, and added 0.03 copolymer of PDMS polymer of 50 copias of viscosity 30 Pa-s = 0.95 mPa, and DPTDPA of 0.03 copy, and it stirred for 10 seconds in the Hauschild™ D102 mixer at 95 rpm, 0.5 copy of emulsion functionality alkoxy (AF2E) of Example 1 was added, and the mixture was stirred for 10 more seconds in this mixer. Subsequently, BTMS of 0.3737 copy was added and this mixture was stirred for 10 more seconds; subsequently — adding Tergitol(trademark)—TMAH surface-active agent of one copy, and 1.5 copias of 0.017 mol % Di undervater glacial—acetic acid solution — this mixture — further — it stirred for 10 seconds and the high solid content o/w emulsion was formed. 0.0372 mol acetic acid of three copias, and three copias of the same were added continuously, and this emulsion was further diluted by stirring this like milk, moved this to the mixer between addition. This silicone emulsion was used for coating the glass plate, which caused this to the Sennco (trademark) cartridge, for 18 hours, the sample was formed on the 254 mm (100 mil.)-thick agar at room temperature under laboratory condition for seven days. The tensile strength of this hardened translucent silicone elastomer was 0.3AMPa (50 psi), 1280% of the maximum elongation, and the Shore A durometer 6. On the glass as a substrate, concrete, the pine tharried out the

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http://www4.ipdl.inp.it/go.ip/cgi-bin/tran\_web\_web.cgi\_cije?atw\_u=http%3A%2F%2Fwww4.ipdl.i... 2010/04/30







pared with nitrogen, and stored into a 50 cc furnace. AFF15 (amino functionality siloxane prepared without using MTM) was gallic within four weeks of storage. About F1 and AFF16, no gelling also took place to the same storage time.

[0137](Example 11) In the polyethylene container, cold blending of siloxane diol (viscosity 0.04 Pa-s in 25 °C), MTM, and the functionality ORGANO siloxane of DP 7-9 was carried out, and the amino functionality siloxane was prepared by making this mixture react for 24 hours. (c) AEAPTMS which uses the following functionality ORGANO siloxanes, (b) Gammex-50 (xylyloxypropyltrimethoxysilane (GPTMS)), (c) gamma-methacryloxypropyl trimethoxy silane (MAPTMS), and (d) gamma-methacryloxypropyltrimethoxysilane (MPTMS) were used. Various functionality ORGANO siloxanes were added to said cold blend by the fixed mole ratio. Table 11 shows the presentation of various functional fluids.

[0138]

Table 12

Two 725-CPH ingredients (part 9) of the 11th table amino functionality siloxanes (a) (b) (c) (d) Siloxane diol DP 7-9 725 746 14AEAPTMS 152 --- GPTMS - 162 --- MATMS --- 17 --- MTMS --- 19-30 12.0 10.2 10.2 10.2019 Each of these functional fluid was evaluated in the elastomeric composition. The addition to the silicone latex compound of a functional fluid was kept on a 1:1 ratio by copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sample prepared using AFF2 (a) showed cohesive failure to glass, and the sample prepared using F2 (b), F3 (c), or F4 (d) showed adhesive failure to glass.

[0140](Example 12) In the polyethylene container, two or more amino functionality siloxanes were prepared by carrying out cold blending of AEAPTMS, MTM, and the hydroxy end siloxane. DP 7-9 indicated for the example 1 (a) using the following siloxane diol, the PDMS ORGANO siloxane which has viscosity 0.04 Pa-s in 25 °C, (b) an average of [average DP 5-7 and 1] — an average of the polyphenyl MECHURISURKORAN ORGANO siloxane of 6.5% of -OH content and (c) average DP 5-7, and 1 — the PORTRIFURISURKORAN ORGANO siloxane of 6.10% of -OH content. These mixtures were made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of the hydroxy end ORGANO siloxane had disappeared. The 12th table shows the presentation of prepared various amino functionality siloxanes.

[0141]

Table 13

12th table amino functionality siloxane component (part) AFF2(AFF17AFF18, dimethylsiloxane diol 74.6 --- phenylmethyl siloxane diol 74.7 --- propylmethyl siloxane diol 74.8 --- 68.3AEAPTMS 15.2 21.08 12.02MTM 10.2 14.5 13.50 14.50 Each of the amino functionality siloxane was evaluated in the elastomeric composition. The addition to said compound of an amino functionality siloxane was kept constant as one copy, the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sample prepared using AFF2 (a) showed cohesive failure to glass, wood, and concrete, and showed mixed deformed using AFF2 (a) of cohesive failure) to aluminum of mill finishing. The sample prepared using AFF17 or AFF18 showed adhesive failure to glass and mill finishing aluminum, and showed cohesive failure to wood and concrete. The physical properties of all the samples are dramatically alike about the durometer, the Shore range of 4-5 is shown, and tensile strength is 0.34 - 0.37MPa.

The maximum elongation was 2060% - 2300%.

[0143](Example 13)

The following experiments were conducted in order to prove the stability of the reserve mixture which consists of an amino functionality siloxane (AFF2), BTMS, hydroxy end JORURGANO siloxane polymer, and DBTDA. The 10-4, Turello MTM mixer was loaded with 5000 copy hydroxy end PDMS polymer (25 cc viscosity 0.04 Pa-s) and AFF2 of 50 copies which prepared said reserve mixture and follows. This mixture was stirred for 30 seconds under the vacuum. adding BTMS of 40 copies, and DBTDA of three copies to this mixture — this mixture — the bottom of a vacuum — it stirred for 30 seconds. When the viscosity of this mixture was observed, there was no big increase in viscosity. 104 g of this reserve mixture was taken at intervals of 60 minutes, and Whip Mix<sup>TM</sup>Mixer of 350mL was loaded. When Tergitol(trademark) TMH-9 surface-active agent of two

copies and DI water of two copies were added to each of these samples and this mixture was stirred for 30 seconds under the vacuum. This mixture was slightly translucent and the o/w emulsion of high solid content was obtained. Subsequently, DI water was stirred for 30 seconds under the vacuum between each addition. [Portions / four / of four copies each]. The sample taken from 1, 2, 3, and 4 showed cohesive failure to glass.

(b) Other experiments were conducted. In order to evaluate whether the amino functionality siloxane has been added however for a long time after emulsifying, hydroxy end PDMS polymer (25 cc viscosity 50 Pa-s) of 5000 copies, BTMS of 40 copies, and DBTDA of three copies were added, and this mixture was stirred for 30 seconds under the vacuum. Tergitol(trademark) TMH-9 of 100 copies, and 6 was added to this mixture, and this mixture was stirred for 30 seconds under the vacuum. Subsequently, when DI water of 150 copies was added and this mixture was stirred under the vacuum for 5 minutes, it was slightly translucent and the o/w emulsion of high solid content was obtained [regular] 60 minutes. Whip Mix<sup>TM</sup>Mixer of 350mL was loaded with these samples, and AFF2 of one copy was added. Subsequently, this mixture was stirred for 30 seconds under the vacuum. To this high solid content silicone emulsion, DI water was stirred for four portions of four copies, respectively, and it added, and stirred for 30 seconds under the vacuum between each addition. The amino functionality siloxane could be added after emulsification of other ingredients of all the 8 hours, and, in addition, giving the cohesive failure of the hardened sealant to glass was found out.

[0144]The Whip Mix<sup>TM</sup> pot of 350mL was loaded with hydroxy end PDMS polymer (viscosity of 25 cc 50Pa, and) of 95.67 copies, and DBTDA of 0.06 copy, and this mixture was stirred for 30 seconds under the vacuum. adding AFF2 of 0.93 copy to this mixture — this mixture — the bottom of 0.08 copy — this mixture — the bottom of a vacuum — further — it stirred for 30 seconds. BTM of 0.70 copy was added to this mixture, and this mixture was stirred for 30 seconds. To this mixture — adding Tergitol(trademark) TMH-9 and DI water of 2.32 copies for 30 seconds, it was slightly translucent — the bottom of a vacuum — further — when this mixture was stirred for 30 seconds, adding 66.28 copies of underwater fluid viscosity of 1000 Pa-s, and this mixture was stirred for 30 seconds. To this mixture — dispersing elements of 1000 Pa-s, and this mixture was stirred for 30 seconds. To this mixture — the mixture — the bottom of a vacuum — further — it stirred for 30 seconds. All the air that filled up the mixture (residual) carried with the silicone emulsion, centrifuged, and was caught was combination was formed on the 2.54 mm (100 mesh) — block film, and it was made to harden under a laboratory condition for 14 days. In tensile strength, 0.84MPa (92 psi) and the maximum elongation were [840% and the Shore A durometer of this silicone emulsion] 22. Furthermore it aged for 18 hours, on the glass as a substrate, concrete, the pine that carried out the painting cloth, and a U.S. Japan cedar, other samples were formed in 90 mm in length, and width of 18 mm, and will be stiffened under a laboratory condition for 14 days. This elastomer showed the adhesive property (cohesive failure mode) excellent in all the substrates.

[Translation done.]